Food Exposures to Lead

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Exposures to lead have emanated from various sources, including food, throughout human history. Occupational and environmental exposures (especially pica) appear to account for much of the identified human disease, however, food-borne exposures deserve further investigation. Lead residues in food can result from: biological uptake from soils into plants consumed by food animals or man, usage of lead arsenate pesticides, inadvertent addition during food processing, and by leaching them improperly glazed pottery used as food storage or dining utensils. Estimates of total dietary exposure should reflect frequency distribution data on lead levels in specific food commodities in relation to the quantities actually ingested by various sample populations to distinguish degrees of risk associated with particular dietary habits. Earlier estimates of average total dietary intake of lead by adults have been reported to range from above 500 μg/day downward with more recent estimates suggesting averages of 200 μg/day or lower. The strengths and weaknesses of these data are discussed along with analytical and sampling considerations.

FDA programs related to food surveillance, epidemiology, and toxicological investigation are briefly described.

Exposures to lead (Pb) have emanated from various sources, including food, throughout human history. While occupational and environmental exposures, especially pica, appear to account for much of the identified human disease, foodborne exposures deserve further investigation (Table 1). Lead residues can result in food from: biological uptake of Pb, including lead-arsenate pesticides, soils, or surface disposition by plants consumed by food animals or man; inadvertent addition during food processing; by leaching from improperly glazed pottery used as food storage or dining utensils. Home food preparation practices also may be potential sources of additional Pb residues in food. Lead in drinking water will also become part of the diet.

Through the years, various estimates of dietary Pb intake have been made, using a variety of sampling techniques and analytical methods. Unfortunately, a basis for valid comparisons to delineate residue trends in specific food commodities appears not to have

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been attained to date, although some surveys of recent origin have been reasonably comprehensive with more adequate methodology.

There are three possible ways to estimate Pb intake from food: measurement of fecal Pb excretion, measurement of Pb content of various food items combined with calculation of a total diet Pb intake based on estimates of food consumption, and measurement of the Pb content of food and beverage collections that specifically duplicate an individual's diet.

Fecal excretion studies, such as those performed by Kehoe (*1*) and by Tompsett and Anderson (*2*), respectively, estimate 300 μg, and 220–400 μg as the normal daily excretion of Pb. Similar studies reported by Tepper (*3*) on 20 American women showed daily fecal excretions of 90–150 μg Pb/day. Studies of fecal Pb excretion performed by Chisholm and Harrison (*4*) included measurements on six control children who had a mean output of 132 μg Pb/day. However, these measurements must be conditioned by the qualification that the degree of absorption from the diet by children is unknown but may be higher than by adults. Fecal Pb measurements in some subjects also may reflect exposures to Pb other than ingestion of foods or water. Lead adsorbed on particulates in air and inhaled is swallowed after being cleared from the respiratory system by the mucociliary apparatus, and thus will be reflected in fecal excretion studies. Fecal excretion studies should be correlated with sampling of drinking water, ambient air in the outer environment and inside the home, and measurements of Pb in street and house dust, aside from measurement of Pb intake in foods as they are ingested.

Table 1. Potential exposures to lead.

Pica	
Ingestion of lead-containing paint chips	
Soil, street, and house dust	
Chewing on newspapers	
Air particulates and/or dust	
Industrial, External, Street, House	Pulmonary absorption, pulmonary clearance and swallowing, inadvertent ingestion.
"Normal" drinking water	
"Normal" dietary intake	
Unusual dietary habits or foods	
Home food preparation	
Water	
House dust	
Paint dust	
Home food storage	
Abrasion of utensils	
Leaching from pottery	

Indirect estimates of total dietary Pb intake have been made throughout recent decades by measuring Pb levels in specific foods and calculating estimated dietary intake of these foods to arrive at estimates of total dietary intake. Historical data on Pb levels in food have been the primary source for such estimates in years past. Weaknesses of such data include lack of knowledge as to whether the foods were sampled in the form that they are usually ingested (e.g., washed and peeled fruits such as apples), although obviously, variations exist as to manner of food preparation before ingestion. Chemical methods also varied, and likely were considerably less accurate at very low Pb concentrations than current methods considered to be acceptable.

A now obsolete estimate was prepared by the Food and Drug Administration (FDA) (*5*) in 1970 (Table 2) by using historical data accumulated over past decades. These data reflect variations in sampling and in analytical methodology; thus these values cannot be said to represent an accurate estimate of current total dietary intake. The estimated average total dietary intake of Pb was 337 μg/day.

In 1972 Pb was included in the FDA Market Basket Survey which involved nationwide sampling of foods representing the average diet of an 18-year-old male, i.e., the individual on a statistical basis eating the highest quantity of food per day. Various food items from the different food classes are purchashed in local market, made up into meals in the proportion that each food item is ingested and cooked or otherwise prepared as they would be consumed. Foods are

Table 2. Preliminary calculation of the lead content of the diet.[a]

Food group[b]	Data source	Lead, ppm	% by weight in total diet	Pb, μg/kg diet
Meat[c]	USDA Survey	0.1	31	31
Cereals	USDA/ HNRD	0.45	25	112.5
Sugars	FDA 1950	0.70[d]	13	91.0
Milk	FDA–CIN	0.06	12	7.2
Fruit and Vegetables	Canada/ FDA	0.40	10	40.0
Nuts and legumes	FDA 1950	1.0	5	50.0
Fats	FDA 1950	0.13	4	5.2
		Total	100	337[e]

[a] Based on "best" estimates as of Dec. 1, 1970.
[b] Beverages and water not included.
[c] No fish or shellfish included. If 10% of the meat is replaced by fish and seafood containing 2 ppm Pb, the Pb from this component becomes 88 μg/kg diet (Instead of 31), the total becomes 394 μg/kg, and the Pb/day becomes 0.59 mg.
[d] Suspiciously high value.
[e] Pb mg/day (based on 1.5 kg diet intake) = (337/1000 x 1.5 = 0.50 mg.

grouped into twelve food classes, then composited and analyzed chemically. While the Market Basket or Total Diet Survey has been in operation by FDA for years to monitor pesticide residues, it is only recently that this technique has been used to measure Pb in the diet. The quantities represented in the Total Diet Survey and the percentage of these food groups in the diet are presented in Table 3. Average Pb content of the 12 food groups and the range of Pb values encountered are shown in Table 4. If all results of analyses are classified as zero, trace, or measured quantity, the calculated daily total dietary intake of Pb is 57.4 μg (Table 5). Because this intake appeared to be low in comparison with previous estimates, we then examined the data and made two sets of conservative assumptions. If it is assumed that all traces detected in fact are at 0.09 ppm, then the calculated dietary intake becomes 159 μg/day (Table 6). If, in addition, all zeroes are assumed to be 0.05 ppm,

Table 3. Daily food intake.

	Food group	Food consumption: Avg. g/day consumed	Food consumption: % of total diet
I	Dairy products	756	26.1
II	Meat, fish, and poultry	290	9.9
III	Grain and cereal products	369	12.6
IV	Potatoes	204	7.0
V	Leafy vegetables	59	2.0
VI	Legume vegetables	74	2.5
VII	Root vegetables	34	1.2
VIII	Garden fruits	88	3.0
IX	Fruits	217	7.4
X	Oils, fats, and shortening	52	1.8
XI	Sugar and adjuncts	82	2.8
XII	Beverages (including water)	697	24.9
	Total	2922	

Table 4. Lead found by food groups.

Food group		Lead found in composites, ppm: Avg.	Lead found in composites, ppm: Range (min.–max.)
I	Dairy products	Trace	0–Trace
II	Meat, fish, and poultry	0.015	0–0.2
III	Grain and cereal products	0.012	0–0.1
IV	Potatoes	0.004	0–0.1
V	Leafy vegetables	0.054	0–0.5
VI	Legume vegetables	0.265	Trace–0.7
VII	Root vegetables	0.131	0–0.8
VIII	Garden fruits	0.108	0–0.3
IX	Fruits	0.031	0–0.3
X	Oils, fats, and shortening	0.015	0–0.2
XI	Sugar and adjuncts	0.008	0–0.1
XII	Beverages (including water)	0.004	0–0.1

the dietary intake rises to 233 μg/day (Table 7).

We then wished to evaluate the total dietary intake of infants and children. In making this estimate, dietary survey data from the United States Department of Agriculture were used in determining foods consumed. Various data on Pb analysis from FDA and the National Canners Association were used. On using the FDA Market Basket data and making the appropriate calcula-

Table 5. Calculated lead intake by food groups.[a]

	Food group	Calculated lead intakes: Avg. ppm	Avg., μg/day	% of total Pb
I	Dairy products	Trace	Trace	Trace
II	Meat, fish, and poultry	0.015	4.4	7.7
III	Grain and cereal products	0.012	4.4	7.7
IV	Potatoes	0.004	0.8	1.4
V	Leafy vegetables	0.054	3.3	5.6
VI	Legume vegetables	0.265	19.6	34.1
VII	Root vegetables	0.131	4.5	7.8
VIII	Garden fruits	0.108	9.5	16.6
IX	Fruits	0.031	6.7	11.7
X	Oils, fats, and shortening	0.015	0.8	1.4
XI	Sugar and Adjuncts	0.008	0.7	1.2
XII	Beverages (including water)	0.004	2.8	4.9
	Total		57.4	

[a] Zeroes and trace amounts not used.

Table 6. Calculated lead intakes.[a]

	Food group	Calculated lead intakes: Avg. ppm	Avg. μg/day	% of total Pb
I	Dairy products	0.028	21.2	13.4
II	Meat, fish, and poultry	0.071	20.2	13.0
III	Grain and cereal products	0.067	24.7	15.6
IV	Potatoes	0.048	9.8	6.2
V	Leafy vegetables	0.126	7.4	4.7
VI	Legume vegetables	0.269	19.9	12.5
VII	Root vegetables	0.162	5.5	3.5
VIII	Garden fruits	0.135	11.9	7.5
IX	Fruits	0.072	15.6	9.8
X	Oils, fats, and shortening	0.057	3.0	1.9
XI	Sugar and adjuncts	0.056	4.6	2.9
XII	Beverages (including water)	0.021	14.6	9.1
	Total		159	

[a] Assumed "trace" = 0.09 ppm.

tions, the average total dietary intake of a 2-year-old child was estimated to be approximately 75 μg/day (Table 8). Correspondingly, if a 6-month-old infant consumes adult table foods, the estimated dietary intake of Pb is approximately 100 μg/day if traces

Table 7. Calculated lead intakes.[a]

	Food group	Calculated lead intakes: Avg. ppm	Avg μg/day	% of total Pb
I	Dairy products	0.062	47.4	20.3
II	Meat, fish, and poultry	0.084	24.4	10.5
III	Grain and cereal products	0.080	29.5	12.6
IV	Potatoes	0.072	14.7	6.3
V	Leafy vegetables	0.135	8.0	3.4
VI	Legume vegetables	0.269	19.9	8.5
VII	Root vegetables	0.172	5.8	2.5
VIII	Garden fruits	0.139	12.2	5.2
IX	Fruits	0.088	19.1	8.2
X	Oils, fats, and shortening	0.080	4.2	1.8
XI	Sugar and adjuncts	0.075	6.2	2.9
XII	Beverages (including water)	0.060	41.8	17.9
	Total		233	

[a] Assumed "Trace" = 0.09 ppm; "zero" = 0.05 ppm.

Table 8. Food items consumed by young children (2-year-old child) and the estimated lead content of these foods.

Food group	% of diet by weight	wt, g	Adult "table foods"[a]: Pb, ppm	Total Pb, μg/day
Dairy products	35.8	560	0.09	45
Meat, fish, and poultry	8.71	136	0.015	2
Grains and cereals	9.61	150	0.012	2
Potatoes	2.37	37	0.004	1
Other vegetables	5.64	88	0.166	15
Fruits	9.29	145	0.05	7
Oils, fats, and shortenings	1.35	21	0.015	<1
Sugars and adjuncts	2.37	37	0.008	<1
Beverages (including water)	24.79	387	0.004	<1
Totals	100.00	1561		73–75

[a] "Market basket" data on lead content of foods, Food and Drug Administration.

are assumed to equal 0.09 ppm levels, and approximately 140 μg/day if additionally all zero levels are assumed to equal 0.05 ppm lead (Table 9). If the 6-month-old infant consumes only infant foods, the estimated

dietary intake of lead would be approximately 120 μg/day (Table 9).

Industry scientists (*6*) analyzed the dietary intake of 333 infants aged from 1 to 12 months and estimated the dietary Pb intake at 93 ± 36 μg/day. By comparison, sampling of Italian infant foods has been reported to estimate a range of 172–289 μg/day for the dietary Pb intake of infants 4–12 months old (*7*). These calculated intakes are all in reasonable accord and suggest that the average dietary intake of Pb of American infants and children up to age 2 is in the range of approximately 100 μg/day. If 200 μg/day is accepted as the average adult Pb intake through total diet, disregarding pulmonary exposure and clearance into the gastrointestinal tract, children up to age 2 then have half the total adult dietary intake. However, children receive a higher exposure to lead per kilogram of body weight as compared to adults. Additionally, some data indicate young animals absorb more Pb (*8, 9*), clinical observations suggest that infants and young children have higher absorption and/or retention of lead from the gastrointestinal tract (*10*).

Accordingly, because evaporated milk and infant formulas comprise much of an infant's diet, further examinations have been made by various scientists. Various data, past and present, exist with respect to lead levels in canned evaporated milk. Data in 1968 on four brands of evaporated milk indicated concentrations of 0.8 ppm; four brands of infant formulas averaged about 0.4 ppm (*11*) Pb. These and other available data indicated the need for further investigation, especially with regard to the solder and flux operations involved with canning evaporated milk. Murphy, Rhea, and Peeler (*12*) reported that market milk samples collected in 1965–66 from 59 cities in the United States averaged 0.047 ppm Pb. More recent data have shown raw milk to average 0.08 ppm lead, while some canned evaporated milk continues to exceed 0.5 ppm lead when levels of 0.2 ppm would have been expected on the basis of concentration of whole milk (*12*). The FDA informed the evaporated milk industry of the need to reduce these levels and the industry instituted a program of investigation and reduction of lead concentrations by making improvements in canning operations and quality control procedures.

In 1972 analyses on samples of four brands by Lamm et al. (*13*) were reported to average 0.36 ppm Pb, whereas 1972–73 levels from 15 samples of three brands reported averaged 0.11 ppm Pb (*14*). Analyses by an approved method subjected to FDA

Table 9. Food items consumed by young children (6-month-old child) and the estimated lead content of these foods.[c]

Food group	6-month-old child					
			Infant foods[b]		Adult "table foods"[c]	
	% of diet by weight	wt, g	Pb, ppm	Total Pb, μg/day	Pb, ppm	Total Pb, μg/day
Dairy products	58.9	803	0.09	72	0.09	72
Meat, fish, and poultry	4.69	64	0.122	8	0.015	1
Grains and cereals	4.40	60	0.200	12	0.012	1
Potatoes	0.62	5	0.004	1	0.004	1
Other vegetables	6.83	93	0.084	8	0.166	15
Fruits	10.49	143	0.104	15	0.05	7
Oils, fats and shortenings	0.59	8	0.015	<1	0.015	<1
Sugars and adjuncts	0.81	11	0.008	<1	0.008	<1
Beverages (including water)	12.91	176	0.004	<1	0.004	<1
Totals	100.00	1363		116–119		97–100

[a] Based on Individual Dietary Survey data from Household Food Consumption Survey, USDA, 1965.
[b] Data supplied by National Canners' Association.
[c] "Market basket" data on lead content of foods, Food and Drug Administration.

audit were carried out by the evaporated milk industry for over 3000 samples during the first 6 months of 1973; the average lead content was found to be 0.125 ppm, the highest level noted in a particular sample being 0.46 ppm (Evaporated Milk Association, unpublished data, 1973). Each sample analyzed consisted of composites of twelve 13-oz. cans or 29 small-size cans (5 1/3 fluid oz.). Additional very recent FDA data on 80 samples, each sample reflecting a composite on 12 cans indicated an average of 0.122 ppm Pb with a range of 0.02–0.37 ppm.

Composite sampling is important because it reflects average consumer exposure. Regulatory considerations presently require that production lots of evaporated milk containing residues of 0.5 ppm or higher are illegal. It is necessary to understand the importance that a guideline has for the quality control cutoff point for industry. Quality control rejection levels have to be lower than the FDA guideline to assure that by the laws of probability FDA sampling of the same lot will not select individual cans with higher than average values and thus condemn the lot by finding excessive levels. We expect from time to time that Pb levels in some individual cans may be higher than desirable, but with the recent processing improvements we feel that this important food commodity will be safe and any further improvements necessary will be implemented.

Commercial infant formulas are prepared from a number of ingredients including several forms of milk. Depending upon availability and price as well as individual company formulations, fresh whole milk, fresh skim milk, condensed whole milk, condensed skim milk, dried milk, or nonfat dried milk may be used. Infant formulas are also prepared from a non-milk base, such as soya bean, for use by individuals allergic to milk protein.

Processing of infant formulas also varies according to individual company procedures. The liquid product, either concentrated or diluted for immediate consumption, may be prepared from fluid milk ingredients or may be reconstituted from dried milk ingredients. The dried infant formulas are normally prepared by drying the concentrated liquid product.

Available information does not indicate a present hazard from lead contamination in the infant formulas. Limited data obtained 5–6 years ago showed values up to 0.4 ppm on the concentrated fluid product. Nine samples of five brands of infant formula concentrated were reported in 1972 (*13*) to average 0.5 ppm lead, whereas six samples of two brands for 1972–73 were reported to show 0.08 ppm lead (*14*). The National Canners' Association reported 15 samples of infant formulas as fed to contain an average of 0.09 ppm lead (*6*). FDA spot analyses of eight samples of four major brands of infant formula concentrates showed an average of 0.09 ppm lead (range 0.06–0.13 ppm) before dilution to make up final formula as ingested. Diluted for consumption, the lead values would generally be reduced by one-half. It should further be kept in mind that infant formula products are balanced nutritionally so that the infant is receiving desirable amounts of calcium, vitamin D, vitamin C, and iron nutrients which if not present in nutritionally adequate amounts, might possibly potentiate the absorption, retention, and toxicity of lead.

Infant foods have been examined to some extent, but FDA plans further investigations of specific commodities in order to identify the needs for further improvement by reducing lead content when necessary and feasible. Certain plant products may be expected to have higher averages than others due to the presence of residues from lead-arsenate pesticide applications and soil exposure and biological uptake. Additional residues from canning operations can be expected to occur on a variable basis, depending upon the character of the food being processed. One survey (*6*) indicated that the weighted average lead content for all strained infant juices studied was 0.38 ppm lead, and FDA sampling of three major brands of orange juice for infants ranged from 0.23 to 0.31 ppm lead. For all other baby foods studied (including dry cereals, baked

goods, meats, fruits, desserts, vegetables and meat dinners, vegetables, and high meat dinners) the weighted average lead content was 0.11 ppm (*6*).

An extensive study by industry was started in 1972 at the recommendation of FDA on the lead contents of a wide variety of canned fruit, vegetable, and meat products. The items are being sampled at various stages of production: raw at the filling point, initially after filling, and after various time increments of storage. The study is not complete at this time, but the levels to date agree in general with those reported by other workers in the field. This study will provide a valuable reference point but it will not reflect the recent special efforts by industry to reduce lead content in canned foods subsequent to the 1972 season.

The FDA also will institute a program to survey Pb in 40 different important staple items of the infant, child, and adult diets. This information, plus that being developed by other workers in the field, will be important in determining the overall levels of lead in the infant and adult diets. It will also be valuable by indicating where technological improvements must be effected, and where it may be important to establish regulatory levels for Pb in foods.

At the same time FDA is carrying out toxicological and epidemiological studies on Pb. This information, in conjunction with the determination of current Pb levels in major food categories of the infant and adult diets, will be taken into account in any future regulations in foods.

We discuss now some of the potential difficulties involved with obtaining accurate sensitive determinations of Pb in various foods.

Chemists have long recognized that the difficulty of obtaining accurate analytical data increases manyfold as the concentration of Pb decreases, particularly at concentrations of less than 1 ppm. Over the years, data relating to the levels of Pb and other trace metals in foods and in biological tissues and fluids have been obtained at considerable effort and cost. Unfortunately, many of these data are of questionable reliability. Specific references which illustrate the lack of accurate analytical methods for Pb determinations or the inadequacy of laboratory performance are scarce. Few analysts are eager knowingly to publish unsatisfactory results. More likely, few recognize errors in analysis resulting from a systematic bias.

An example of variation between laboratories is the result of a recent Environmental Protection Agency–National Bureau of Standards interlaboratory study (*15*). The samples examined by the participants in this study will be issued by the NBS as Standard Reference Materials (SRM). One of these SRM is fly ash. The chromium content of the fly ash SRM is quite high, with a probable certified value of 125 ± 10 ppm. Even at this high concentration, the results submitted by 23 laboratories were poor; 20 of the 23 values were incorrect, although the analytical mean was the same as the probable certified value. Of the three analytical techniques employed, atomic absorption spectroscopy, neutron activation analysis, and optical emission spectroscopy, it is noteworthy that the most frequently used technique, atomic absorption spectroscopy, gave the widest range of values reported. The scattered performance cannot be explained on the basis of low chromium content or poor sensitivity.

Accuracy worsens as the trace metal level decreases. This was demonstrated by the results obtained for the determination of cadmium in the fly ash SRM. The mean of the 12 values reported was 3.9 ppm, compared with the probable certified value of 1.45 ± 0.06 ppm. All 12 results were incorrect, the largest discrepancies being the results by atomic absorption spectroscopy. Since both techniques used (atomic absorption and isotope dilution spark source mass spectroscopy) are quite sensitive for cadmium, neither low concentration nor poor sensitivity account for the inaccuracy of the results.

The determination of Pb in the range below 1 ppm is possibly even more suscepti-

ble to error than the two examples given above, in part because the sensitivity of the common techniques is relatively poor for Pb analysis. Keppler et al. (*16*) published in 1970 the findings of an extensive interlaboratory study of the determination of Pb in blood. Over 60 laboratories participated in each of two studies, and each laboratory used its method of choice. Although many common techniques were employed (e.g., spectrophotometry and colorimetry, polarography, atomic absorption, and spectrography-film and direct reader) only the spectrophotometric and atomic absorption techniques were used by enough laboratories to give realistic reliability estimates. Unacceptable results were reported by 60% of the participants involved in both studies. One-half of the results were judged unacceptable in each of the two studies. Statistical analysis showed no significant differences by type of laboratory or by procedure used. Interestingly, many of the better results in the second study were obtained by spectrophotometry while most of the poorest were obtained by laboratories using atomic absorption or polarography. The results of an unpublished interlaboratory study of the determination of Pb in evaporated milk (Evaporated Milk Association, unpublished data, 1972) also support the conclusion that most common methods of Pb analysis are generally inadequate.

The ubiquity of Pb greatly magnifies the difficulties involved in its estimation. There are a number of potential sources of error in the determination of Pb and other trace elements in foods (*17*). All phases of an analysis including sampling, sample preparation, and measurement, must be conducted with exceptional care (Table 10). Sampling, in the broadest sense, is not within the scope of this discussion although it is obviously of great importance in the interpretation of the significance of the data. Errors directly related to the analysis can be either positive or negative and can originate with the sample preparation and with the measurement.

Both contamination and loss of Pb can occur in the process of obtaining a representative sample. Most samples cannot be assumed to be homogeneous, and the processes of cutting, milling, grinding, and blending are likely sources of contamination, although the use of stainless steel in the construction of comminuting equipment has largely minimized this source of error. Sample contamination frequently arises from the other manipulations necessary in an analysis, such as treatment with reagents (acids, water, ashing-aids, etc.), apparatus (glassware, ashing vessels, ovens, and furnaces) and the laboratory environment (air, dust, etc.). Trace metal loss during the destruction of the sample matrix is a well known source of error. Lead can be lost by volatilization or by retention in the ashing vessel. When poor recoveries occur, it is usually found that dry-ashing was the technique used to mineralize the sample. Dry-ashing is most often employed because it can be used to obtain the large amounts of sample

Table 10. Probable sources of error for common analytical methods to determine lead in foods.

Operation	Error	Origin
Sampling	+	Surface contaminants
		Contamination during homogenization
Sample mineralization and manipulations		Dry ashing losses
		Reagent contamination (air, water, acids)
	+	Surface contamination from apparatus
		Dry ashing contamination from ashing vessels
Measurement AAS	+	Nonspecific absorption (scattering)
	−	Inadequate sensitivity
Polarography	+	Chemical interferences (Sn, Tl)
	−	Inadequate sensitivity
Spectrophotometry	+	Spectral interferences (Sn, Bi, Tl)
	−	Inadequate sensitivity

required to compensate for the relatively poor sensitivity of the common analytical techniques. Moderate ashing temperatures and the use of so-called ashing aids are considered necessary by most analysts to obtain reliable Pb values. Temperatures up to 450° C. have been found to allow quantitative recovery of the Pb in most cases with losses occurring more frequently as the ashing temperature is increased above this value. Magnesium nitrate and sulfuric acid are two of the many ashing aids which are effective in minimizing Pb losses during mineralization.

Frequently the method of measurement is overlooked as a source of error. Interferences due to the presence of other metals are common in absorption spectrophotometry and polarography but rare in atomic absorption measurements. Matrix effects are a very real problem for the latter technique, especially when very low levels of Pb are to be determined. Certainly where a Pb concentration of 0.5–1 μg/ml of aqueous solution produces only 1% absorption by atomic absorption spectrophotometry there is a tendency to attempt to extract more information than is consistent with the capabilities of the technique. For example, if 25-g sample containing 0.1 ppm Pb is ashed and the residue dissolved in 50 ml of dilute mineral acid, the absorption due to Pb constitutes only 0.1%. Matrix effects such as nonspecific absorption (e.g., light scattering) can give rise to signals which are of the same order of magnitude or greater than that due to Pb, depending on the nature and amount of ash, the flame type, and stoichiometry.

Spectral interferences by tin, bismuth, and thallium cause positive errors in the dithizone-spectrophotometric method for Pb. The half-wave potentials of tin and thallium are sufficiently close to that of Pb to cause a false positive peak for Pb in the polarographic estimation of Pb.

These examples are meant to be only illustrative of the lack of reliable methodology. The need for better and more sensitive techniques such as anodic stripping, voltammetry, and "nonflame" atomic absorption spectrophotometry are finding their way into laboratories. Since analytical methods all depend upon calibration with suitable standards, the availability of standard reference materials is extremely important. Standards are needed which better approximate the organic matrices of the various samples and trace metal levels usually encountered. The NBS bovine liver SRM is a recent example. Recent and future cooperative efforts of the various government agencies may accelerate the expensive process of providing more standard reference materials.

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